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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant: Pamela Whitfield et al.
Serial No: 10/558,445
Filed: November 6, 2006
Title: Lithium Metal Oxide Electrodes for Lithium Cells and Batteries
Examiner: Fatou G. Maiga **Group Art Unit:** 1745
Docket No.: 11495-1

**Hon. Commissioner of Patents
and Trademarks
Washington, D.C. 20231
U.S.A.**

DECLARATION UNDER 37 CFR 1.132

I, Isobel Davidson, residing at 1569 Delia Crescent, Orleans, Ontario, K4A 2X7, hereby declare as follows:

1) that I am a co-inventor of the above-identified patent application and a co-author of the publication entitled "Anisotropic Peak Broadening in $\text{Li}_{1.2}\text{Mn}_{0.4}\text{Ni}_{0.3}\text{Co}_{0.1}\text{O}_2$ Charged to High Voltage" which was printed in the Programme and Extended Abstracts of the 3rd Lithium Battery Discussions – Electrode Materials, Bordeaux-Arachon, France 22-27 May 2005, attached hereto as Exhibit "A",

with P.S. Whitfield, S. Niketic, and Y. Le Page as co-authors;

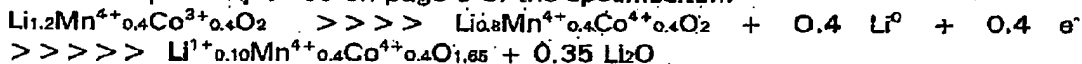
- 2) that I qualify as a person skilled in the art of the present invention, as evidenced by the copy of my curriculum vitae which includes a list of my publication credits, attached hereto as Exhibit "B"
- 3) that for clarification purposes, we have voluntarily amended the structural formula of the claimed materials in claims 1 and 6 to recite amounts of Li, Mn and M in terms of the variables x, y and z, respectively, based upon original Tables 2 and 3. Similarly, in claim 6, the structural formula of the starting materials now recites the amounts of Li, Mn and M in terms of the variable x', y' and z', respectively. It is believed that since the amounts of Li, Mn and M are taken from original Tables 2 and 3, or are based on values determined by single mathematical calculations, and attested to herein, that no "new matter" has been presented.
- 4) that more specifically, regarding the general formula of the claimed final materials of the invention of $\text{Li}_x\text{Mn}_y\text{M}_{1-y}\text{O}_2$ to simplify matters,

we have changed the formula in claim 1 to $\text{Li}_x\text{Mn}_y\text{M}_z\text{O}_2$, wherein $0 < x \leq 0.24$, $0 < y < 1$, $z = 0.46$ to 0.86 and $y + z = 0.94$ to 0.99 , manganese is in the 4+ oxidation state and M is one or more transition metal or other cations, but is not solely Ni or Cr.

In this formula, the value of y is the same as the value of y in the original Description, and the values of x and z are based upon the Description of the reactions on page 9 and on simple mathematical calculations, based upon the data in original Tables 2 and 3. It is noted that the value of x has simply been made more precise based upon the mathematical calculations.

More specifically, the Examiner should appreciate that the difficulty in defining the formula of the material we are trying to claim is that the material is formed in situ in an electrochemical cell on deep charging of the starting material. Deep charging occurs in two steps: first loss of lithium ions balanced by increases in the oxidation state of the M and secondly via loss of Li_2O without further changes in the oxidation state of Mn or M. At the end of deep charging both the lithium and oxygen content have decreased relative to the formula of the starting material.

This concept is explained on page 9 of the specification:



and it should be recognized that $\text{Li}^{1+}_{0.10}\text{Mn}^{4+}_{0.4}\text{Co}^{4+}_{0.4}\text{O}_{1.65}$ can be equivalently described (normalized) as $\text{Li}_{0.12}\text{Mn}^{4+}_{0.495}\text{Co}^{4+}_{0.495}\text{O}_2$

As illustrated above, in one approach, the amounts of lithium and oxygen are reduced and the amounts of Mn and M (in this case $\text{M}=\text{Co}$) remain the same. In another equivalent approach, the formula can be normalized to two stoichiometric units of oxygen by a simple mathematical calculation.

We have adopted the latter approach.

By way of further explanation, the Examiner is referred to the following data presented in the form of expanded Tables 2 and 3, which include "normalized" values for x , y and z , based upon simple mathematical calculations well known to persons skilled in the art using information in original Tables 2 and 3.

The "normalization" of the composition as obtained at the end of charge is calculated by reference to the data in Tables 2 and 3. For example in Table 2, the initial compositions are solid solutions of in 1:1 ratios Li_2MnO_3 and $\text{LiNi}_{1-x}\text{Co}_x\text{O}_2$. Such compositions can be equivalently described as $\text{Li}_{1.2}\text{Mn}_{0.4}\text{Ni}_{0.4-x}\text{Co}_x\text{O}_2$ where x varies from 0 to 0.4. At the end of charge all the metal cations are charged to the 4+ oxidation state. The lithium content of the charged materials is listed in the Tables 2 and 3. The oxygen content of the charged materials is calculated from the final stoichiometry of lithium recognizing that the content of other metals is unchanged on charging. The formula can then be normalized to an oxygen stoichiometry of two.

Similarly for the data in Table 3, at the end of charge all metals are in their highest oxidation state.

For example if we consider the final entry in Table 2, the nominal stoichiometry is $\text{Li}_{1.2}\text{Mn}_{0.4}\text{Co}_{0.4}\text{O}_2$, the more precise lithium stoichiometry determined by atomic absorption spectroscopy is 1.172. The lithium stoichiometry of the final product as listed in the Table 2 in the disclosure is 0.20. The content of Mn and Co are not changed by charging and both Mn and Co are in +4 oxidation state at the end of charge. Consequently the stoichiometry of the product at the end of charge is $\text{Li}_{0.2}\text{Mn}_{0.4}\text{Co}_{0.4}\text{O}_{1.70}$ with the oxygen content being defined by the stoichiometry of the metals and oxygen being in the -2 oxidation state.

I.E. Total metals: $(0.2 \times 1) + (0.4 \times 4) + (0.4 \times 4) = 3.40$, consequently the oxygen stoichiometry is $3.40/2 = 1.70$.

Re-writing or normalizing the formula to an oxygen stoichiometry of 2 yields the formula $\text{Li}_{0.24}\text{Mn}_{0.47}\text{Co}_{0.47}\text{O}_2$.

I.E. Lithium content $0.2/1.70 \times 2.0 = 0.24$
Mn and Co content $0.4/1.70 \times 2.0 = 0.47$

Table 2 with calculated compositions for end-of-charge

Initial Nominal Composition	Initial Li Content (AA)	Final Li Content	Final Composition	Normalized Final Composition
$\text{Li}_{1.2}\text{Mn}_{0.4}\text{Ni}_{0.4}\text{O}_2$	1.162	0.32	$\text{Li}_{0.32}\text{Mn}_{0.4}\text{Ni}_{0.4}\text{O}_{1.76}$	$\text{Li}_{0.36}\text{Mn}_{0.46}\text{Ni}_{0.46}\text{O}_2$
$\text{Li}_{1.2}\text{Mn}_{0.4}\text{Ni}_{0.3}\text{Co}_{0.1}\text{O}_2$	1.146	0.20	$\text{Li}_{0.2}\text{Mn}_{0.4}\text{Ni}_{0.3}\text{Co}_{0.1}\text{O}_{1.70}$	$\text{Li}_{0.24}\text{Mn}_{0.47}\text{Ni}_{0.36}\text{Co}_{0.12}\text{O}_2$
$\text{Li}_{1.2}\text{Mn}_{0.4}\text{Ni}_{0.2}\text{Co}_{0.2}\text{O}_2$	1.174	0.20	$\text{Li}_{0.2}\text{Mn}_{0.4}\text{Ni}_{0.2}\text{Co}_{0.2}\text{O}_{1.70}$	$\text{Li}_{0.24}\text{Mn}_{0.47}\text{Ni}_{0.24}\text{Co}_{0.24}\text{O}_2$
$\text{Li}_{1.2}\text{Mn}_{0.4}\text{Ni}_{0.1}\text{Co}_{0.3}\text{O}_2$	1.158	0.09	$\text{Li}_{0.09}\text{Mn}_{0.4}\text{Ni}_{0.1}\text{Co}_{0.3}\text{O}_{1.65}$	$\text{Li}_{0.11}\text{Mn}_{0.49}\text{Ni}_{0.12}\text{Co}_{0.36}\text{O}_2$
$\text{Li}_{1.2}\text{Mn}_{0.4}\text{Co}_{0.4}\text{O}_2$	1.172	0.20	$\text{Li}_{0.2}\text{Mn}_{0.4}\text{Co}_{0.4}\text{O}_{1.70}$	$\text{Li}_{0.24}\text{Mn}_{0.47}\text{Co}_{0.47}\text{O}_2$

Table 3 with calculated compositions for end-of-charge

Initial Nominal Composition	Final Li Content	Final Composition	Normalized Final Composition
$\text{Li}_{1.2}\text{Mn}_{0.2}\text{Ti}_{0.2}\text{Ni}_{0.2}\text{Co}_{0.2}\text{O}_2$	0.20	$\text{Li}_{0.20}\text{Mn}_{0.2}\text{Ti}_{0.2}\text{Ni}_{0.2}\text{Co}_{0.2}\text{O}_{1.70}$	$\text{Li}_{0.24}\text{Mn}_{0.24}\text{Ti}_{0.24}\text{Ni}_{0.24}\text{Co}_{0.24}\text{O}_{2.0}$
$\text{Li}_{1.2}\text{Mn}_{0.4}\text{Ni}_{0.2}\text{Co}_{0.1}\text{Al}_{0.1}\text{O}_2$	0.28	$\text{Li}_{0.28}\text{Mn}_{0.4}\text{Ni}_{0.2}\text{Co}_{0.1}\text{Al}_{0.1}\text{O}_{1.69}$	$\text{Li}_{0.33}\text{Mn}_{0.47}\text{Ni}_{0.24}\text{Co}_{0.12}\text{Al}_{0.12}\text{O}_{2.0}$
$\text{Li}_{1.158}\text{Mn}_{0.316}\text{Ni}_{0.263}\text{Co}_{0.263}\text{O}_2$	0.17	$\text{Li}_{0.17}\text{Mn}_{0.316}\text{Ni}_{0.263}\text{Co}_{0.263}\text{O}_{1.77}$	$\text{Li}_{0.19}\text{Mn}_{0.36}\text{Ni}_{0.30}\text{Co}_{0.30}\text{O}_{2.0}$
$\text{Li}_{1.138}\text{Mn}_{0.270}\text{Ni}_{0.297}\text{Co}_{0.298}\text{O}_2$	0.06	$\text{Li}_{0.06}\text{Mn}_{0.270}\text{Ni}_{0.297}\text{Co}_{0.298}\text{O}_{1.76}$	$\text{Li}_{0.06}\text{Mn}_{0.31}\text{Ni}_{0.34}\text{Co}_{0.34}\text{O}_{2.0}$

$\text{Li}_{1.059}\text{Mn}_{0.118}\text{Ni}_{0.414}\text{Co}_{0.414}\text{O}_2$	0.10	$\text{Li}_{0.10}\text{Mn}_{0.118}\text{Ni}_{0.414}\text{Co}_{0.414}\text{O}_{1.84}$	$\text{Li}_{0.10}\text{Mn}_{0.12}\text{Ni}_{0.43}\text{Co}_{0.43}\text{O}_{2.0}$
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Regarding the formula for the starting materials, included in claim 6, it will be appreciated that clarification is also required. Accordingly, we have adopted the following formula $\text{Li}_x\text{Mn}_y\text{M}_z\text{O}_2$, wherein $x' = 1.059$ to 1.2 , $0 < y' < 1$, $z' = 0.4$ to 0.828 and $y' + z' = 0.8$ to 0.95 (value for y' is as original and values for x' and z' taken from Tables 2 and 3).

- 5) that accordingly, this supporting material be taken into consideration in the prosecution of this application.
- 6) that the publication described in 1) above includes the "crystallographic" structural data relevant to the materials described in the claims, and wherein Figures 1, 3 and 4 show quite clearly that a layered crystallographic structure is maintained after the initial charging to 4.6 volts.

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that wilful false statements and the like so made are punishable by fine or imprisonment or both, under Section 1001 of Title 18 of the United States Code and that such wilful false statements may jeopardize the validity of the application or any patent issued thereon.

Isobel Davidson
Isobel Davidson

Date: March 14, 2008

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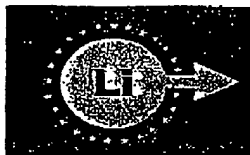
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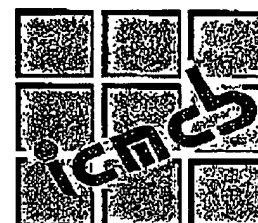
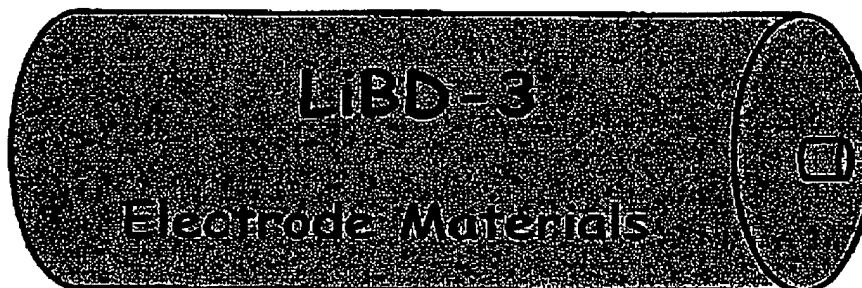
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EXHIBIT "A"

LITHIUM BATTERY DISCUSSIONS - ELECTRODE MATERIALS -



ERICSSON



PROGRAMME & EXTENDED ABSTRACTS

Bordeaux-Arcachon, France
22 - 27 May 2005

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Anisotropic Peak Broadening in $\text{Li}_{1.2}\text{Mn}_{0.4}\text{Ni}_{0.3}\text{Co}_{0.1}\text{O}_2$ Charged to High Voltage

Pamela Whitfield, Svetlana Niketic, Yvon Le Page and Isobel Davidson

Institute for Chemical Process and Environmental Technology, National Research Council Canada, 1200 Montreal Road, Ottawa ON K1A 0R6, CANADA

A number of systems consisting of Li_xMnO_2 solid solutions with NiO [1] and $\text{LiNi}_{1/2}\text{Mn}_{1/2}\text{O}_2$ [2] have recently been the subject of interest. Their stability at high voltages and extremely good reversibility even when most of the lithium is removed on charge is intriguing. The subject of this study, $\text{Li}_{1.2}\text{Mn}_{0.4}\text{Ni}_{0.3}\text{Co}_{0.1}\text{O}_2$ has been shown to be just such a material [3,4], and an ex-situ X-ray diffraction study was undertaken to study its structural behaviour during cycling.

A few papers have been published examining the structural behaviour of Li_xNiO_2 and $\text{Li}_x\text{Ni}_{1-x}\text{Co}_x\text{O}_2$ where x is close to 0. This has either been achieved by holding at moderately high voltages for extended periods [5], or charging galvanostatically to very high voltages [6,7].

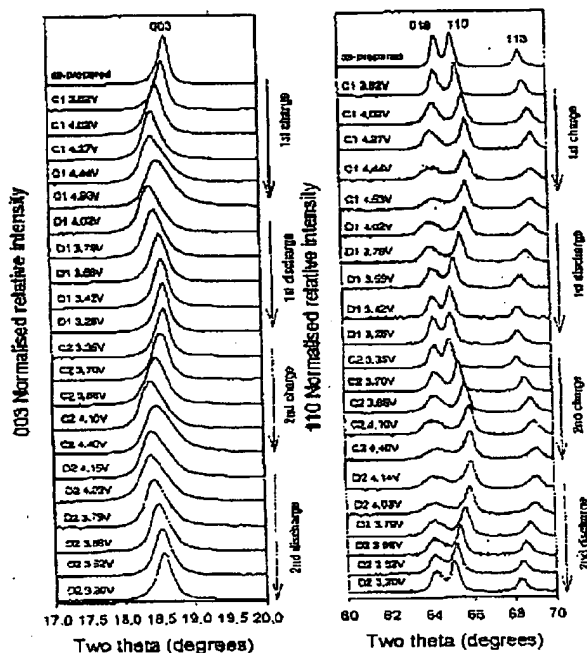


Figure 1. Ex-situ X-ray diffraction data from $\text{Li}_{1.2}\text{Mn}_{0.4}\text{Ni}_{0.3}\text{Co}_{0.1}\text{O}_2$ during the first two cycles between 2.0 and 4.6V.

The $\text{Li}_{1.2}\text{Mn}_{0.4}\text{Ni}_{0.3}\text{Co}_{0.1}\text{O}_2$ was produced using a sucrose-based process previously described [4]. Electrodes consisting of active material, Super S and Kynarfex binder on aluminium foil, were charged/discharged to the desired voltage versus lithium in a 2325 coin cell using 1M LiPF_6 in 1:1 EC:DMC

electrolyte. The cell was disassembled in a glove box, and the active material washed and centrifuged repeatedly with dry DMC. After drying under vacuum, X-ray diffraction patterns were obtained using a parallel-beam Bruker D8 instrument using $\text{CuK}\alpha$ radiation. The small amount of sample was mounted onto a silicon zero-background holder.

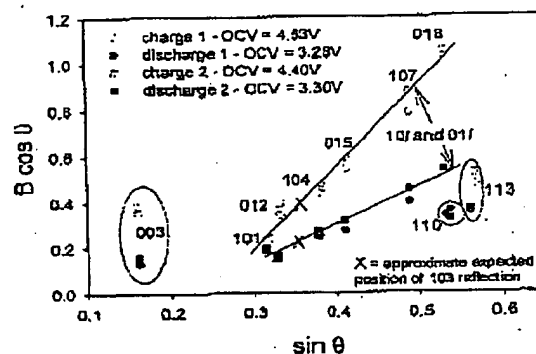


Figure 2. Williamson-Hall plot of $\text{Li}_{1.2}\text{Mn}_{0.4}\text{Ni}_{0.3}\text{Co}_{0.1}\text{O}_2$ at the end of charge and discharge.

The reversible development of anisotropic peak broadening on charge is very noticeable (Figure 1). The hkl -dependence is shown graphically in Figure 2 in the form of a Williamson-Hall plot [8] from data at the end of charge and discharge during the first two cycles. On charge there was significant broadening of the 10/ and 01/ reflections, whilst the a - b plane dependent 110 reflection was not affected. Figure 2 also shows that the microstructural changes in the material are highly reversible. This type of hkl -dependent broadening in 10/ and 01/ has been attributed to stacking defects in highly charged materials [5]. However, the behaviour of the 003 reflection relative to the 10/ and 01/ reflections is not consistent with stacking faults in a homogeneously delithiated structure. In this case, the 00/ reflections should exhibit the largest broadening. The behaviour of the 003 is more consistent with varying residual lithium content throughout the grains with no stacking disorder, leading to a distribution of interlayer spacing. This model leads to line broadening which follows the relationship:

$$2q \times \delta \times c^* \times \cos(c^* \cdot R^*)$$

where c^* is the c reciprocal space vector, R^* is the reciprocal vector of interest, q is a small constant, and δ is the distribution of lithium contents in the material $\text{Li}_{x_0-\delta}\text{Mn}_{0.4}\text{Ni}_{0.3}\text{Co}_{0.1}\text{O}_2$ where $x_0 - \delta < x_1 < x_0 + \delta$. This

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model would give broadening of 003 similar to 113 and that interpolated for 103, which is close to the observed behaviour. In reality, the real material will not be ideal, but will exhibit both stacking faults and compositional non-uniformity. However the observations suggest that stacking faults are not the dominant effect in producing the anisotropic broadening in this material when the lithium layers are seriously depleted.

The ability of Topas [9] to convolute additional functions into the peak profiles enabled full-pattern fittings to be performed. To gauge state-of-charge in electrodes, it was necessary to estimate the proportion of isolated starting material. Consequently, the refined structure of the starting material was used to simultaneously perform quantitative analysis.

A simple 2-phase refinement using the O3 structure without modelling the peak anisotropy as shown in Figure 3 leads to serious misfits and no residual starting material. This has consequences due to inaccurate extraction of peak intensities for the quantitative analysis and the calculated bond lengths. Figure 4 shows that sensible results may be obtained using an O3 structure with a small residual amount of starting material (7.8 wt%) when the peak intensities can be accurately extracted. Satisfactory fits were obtained, and the refined bond length is consistent with values expected for highly oxidised material having a composition close to $\text{Mn}^{4+}_{0.3}\text{Ni}^{4+}_{0.375}\text{Co}^{4+}_{0.125}\text{O}_{2-x}$.

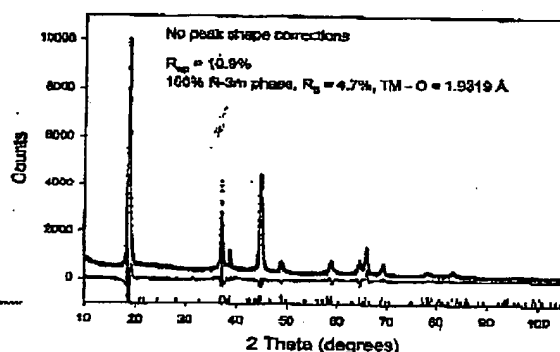


Figure 3. Rietveld difference plot for $\text{Li}_{1.2}\text{Mn}_{0.4}\text{Ni}_{0.3}\text{Co}_{0.1}\text{O}_2$ at the end of 1st charge without any peak-shape corrections.

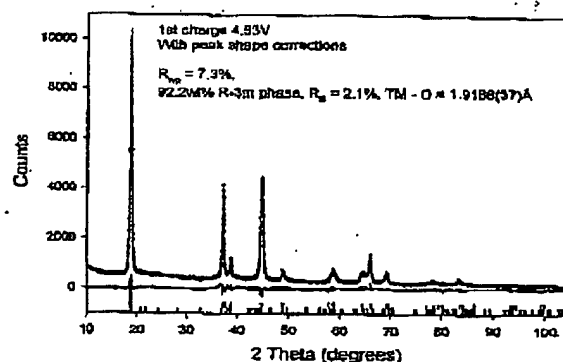


Figure 4. Rietveld difference plot for $\text{Li}_{1.2}\text{Mn}_{0.4}\text{Ni}_{0.3}\text{Co}_{0.1}\text{O}_2$ at the end of 1st charge with peak-shape corrections to fit the anisotropy.

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EXHIBIT "B"

Dr. Isobel J. Davidson

Current Position: *Competency Group Leader Ceramic Materials and Project Leader for Li-ion Battery Materials Development at National Research Council Canada*

Expertise:

- *Energy storage and conversion devices*
 - *Design and evaluation of functional materials for high energy density batteries and solid oxide fuel cells*
 - *Evaluations of the relationships between the structure and composition of electrode materials and their electrochemical performance and thermal safety*
- *Solid-state and inorganic chemistry*
 - *Crystallographic studies of polycrystalline materials*
 - *Inorganic synthesis by conventional and soft chemical techniques*
 - *Evaluation of electronic and magnetic properties*

Education:

- *Ph.D. Solid State Chemistry, McMaster University, Hamilton, Ontario.*
 - *Thesis: Synthesis, Structure and Properties of Selected Lithiated Transition Metal Oxides.*
- *M.Sc. Inorganic Chemistry, McMaster University, Hamilton, Ontario.*
 - *Thesis: A Structural Study of LiMO_2 ($M=\text{Ru}$, Os and Ir) by X-ray and Neutron Diffraction*
- *B.Sc. Honours Chemistry, McMaster University, Hamilton, Ontario.*
 - *Thesis: Neutron Scattering in Solid CH_3D .*

Employment History:

September 1987 to Present - Research Officer – National Research Council Canada

- *Project Leader for lithium ion battery materials development (1990 – present)*
- *Senior Research Officer / Group Leader (1999 – present)*
- *Program manager for Federal Program on Plug-In Hybrid Electric Vehicles (2007 – present)*

June 1983 – August 1987 - Research Chemist – Ballard Research Inc. B.C.

- *Proprietary research on rechargeable Li/SO_2 batteries.*
- *Structured research programs for technologists and student research assistants.*
- *Resolved material compatibility issues, identified limiting features in battery performance and optimized cell chemistry and format – U.S. patent issued.*

Honours and Awards:

- *NRC awards for generation of substantial royalties resulting from the licensing of ICPET lithium ion battery technology in 2001, 1999 and 1996.*
- *NRC Outstanding Achievement Award for Contributions to Clients - 2001.*

Professional Activities:

- Member, Electrochemical Society
- Technical reviewer for IRAP and Technology Partnerships Canada
- Technical reviewer for NSERC Strategic Grants and United Kingdom - Engineering and Physical Sciences Research Council Grants

Publications

Pamela Whitfield, Ali Abouimrane, and Isobel Davidson, "Investigation of Plastic Crystals based on Succinonitrile as Solid Electrolytes for Lithium Batteries", 55th Denver X-Ray Conference, Denver, CO, USA, 7-11 August 2006, *Advances in X-ray Analysis*, volume 50 (*in press* 2007).

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C. Bock, B. MacDougall, Y. Yoo, I-M. Hung, S. Koutcheiko and I. Davidson, "Applications of Nano-Materials in Fuel Cells", Nano-materials Crossroads 2004, Boucherville, Quebec, October 19, 2004.

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I. J. Davidson, "*Electrochemical Power Source R&D at ICPET*", DRDC workshop on Electrochemical Power Sources for the Canadian Forces March 19, 2003.

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I. Davidson, "*Neutrons and Battery-related Materials*", NRC Neutron Scattering Summer School, Chalk River Laboratories, June 22, 2000.

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J. Xie, Y. Yoo, I. Davidson, and D. Ghosh, "*Oxidation Behavior of Metallic Interconnects for IT-SOFC and Area Specific Resistance of Oxide Scales*", presented in a poster presentation at NRC workshop on Ceramic Materials for Fuel Cells in Ottawa, Feb. 20, 2004.

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